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THE THEORY AND CONSTRUCTION OF A LIQUID HELIUM CRYOGENIC PUMP

GEORGE M. LA CHANCE

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George M. LaChance

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by

George M. LaChance

Lieutenant, United States Navy

Submitted in partial fulfillment of
the requirements for the degree of

MASTER OF SCIENCE
IN
MECHANICAL ENGINEERING

United States Naval Postgraduate School
Monterey, California

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ABSTRACT

Cryogenic pumping is a means of maintaining a high vacuum with large gas in-leakage rates. This is accomplished by the condensation and freezing of the gas molecules onto a surface that is maintained at a temperature well below the freezing point of the gas. A liquid helium filled "batch process" cryogenic pumping system was constructed and instrumented. The vacuum chamber and roughing system were obtained by the modification and redesign of an existing vacuum furnace. The theory of cryogenic pumping is presented and an approach developed for the evaluation of the cryopump performance.

ACKNOWLEDGEMENTS

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TABLE OF SYMBOLS

A	surface area	cm^2
Eb	emmisive power	watts cm^{-2}
F	shape factor	---
L	length	cm
M	molecular weight	gm
N	molecule flow rate	sec^{-1}
No	Avagadro's Number	mol^{-1}
n	molecules per unit volume	cm^{-3}
P	pressure	microns Hg
Q	throughput rate	micron liters sec^{-1}
q	heat rate	watts sec^{-1}
R	universal gas constant	micron liters $°K^{-1}$ $gmole^{-1}$
Ro	universal gas constant	ergs
S	pumping speed	liters sec^{-1}
T	temperature	°K
t	time	sec
V	volume	liters
v	molecule speed	cm sec^{-1}
γ	capture (or sticking) coefficient	---
ϵ	emissivity	---
λ	mean free path	cm
μ	coefficient of viscosity	gm cm^{-1} sec^{-1}
σ	Stefan-Boltzmann Constant	watts cm^{-2} $°K^{-4}$

TABLE OF SUBSCRIPTS

c	conditions of vapor in equilibrium with solid phase
i	conditions of induced in-leakage gas
o	conditions within chamber without cryopumping and induced in-leakage
r	conditions of residual in-leakage gas

1. Introduction.

The recent interest in space simulation has led to the requirement of a pump that will generate and maintain a high vacuum with large in-leakage rates. The cost to accomplish this with mechanical and diffusion pumps is prohibitive, due to the very large size of equipment required to handle the large volumes of gases at very low pressures which must be pumped to maintain the high vacuum. In the past few years, the cryogenic pumping method has been investigated as a means of obtaining the desired results at much less cost and size. /1, 15/

Cryogenic pumping, hereafter called cryopumping, produces a vacuum by the condensation and solidification of a gas or vapor onto a cold surface. It will pump any gas whose condensate vapor pressure, at the temperature of the surface, is less than the required pressure of the system.

The objectives of this thesis were: the modification and redesign of a 40 inch diameter vacuum furnace system to be used as the vacuum or space simulation chamber, the installation of heat shields to shield the cryopanel from thermal radiation from the chamber walls, the design and installation of the cryopanel upon which the gas condensation occurs, and the development of the theory for an evaluation of the cryopump.

2. Theory of Cryopumping.

In a high vacuum, the molecules of gas are relatively far apart and the kinetic theory of gases and the perfect gas law may be assumed to apply. Within a closed system the number of molecules passing through a unit area per unit time may be derived from kinetic theory /18/:

$$\dot{N} = \frac{1}{4} n\bar{v} \tag{1}$$

where n is the number of molecules per unit volume, and $\bar{v}$ is the mean molecular velocity, which can be related to the gas properties /18/:

$$\bar{v} = \left(\frac{8R_oT}{\pi M}\right) \tag{2}$$

By the use of (1), (2), and the perfect gas law, the fraction of molecules within the chamber that strike the cryopanel of area A per unit time is:

$$\frac{\dot{N}}{N} = \frac{\frac{A}{4}\left(\frac{PN_o}{RT}\right)\left(\frac{8R_oT}{\pi M}\right)^{1/2}}{PVN_o/RT} = \frac{A}{V}\left(\frac{R_oT}{2\pi M}\right)^{1/2} \tag{3}$$

But of the total number of molecules per second which strike the panel, only a fraction, δ, stick to the panel and condense, while the more energetic molecules lose some of their energy and rebound. The fraction of the molecules within the chamber that stick to the cryopanel per second is:

$$\frac{\dot{N}_p}{N} = \delta\frac{\dot{N}}{N} = \delta\frac{A}{V}\left(\frac{R_oT}{2\pi M}\right)^{1/2} \tag{4}$$

where δ is called the capture coefficient.

Since, by the kinetic theory, the pressure is proportional to the number of molecules per unit volume, then the rate of pressure drop due to cryopumping is:

$$\dot{P} = - \frac{P \delta A}{V} \left(\frac{R_o T}{2 \pi M} \right)^{1/2} \qquad (5)$$

The rate of pressure rise due to an in-leakage rate, $P_r \dot{V}_r$, is

$$\dot{P} = \frac{P_r \dot{V}_r}{V} \qquad (6)$$

assuming that the temperature of the inleaking gas, T_r , is equal to the average temperature of the gas within the chamber, T . This is nearly the case, as at low pressure the conduction and convection from the gas are negligible, and the time for the gas to cool by radiation is long compared to the time for the gas to be pumped. This assumption, usually made /15/, is conservative as the rate of pressure rise would be reduced by a factor of $\left(\frac{T}{T_r} \right)$.

The total rate of change of pressure is equal to the sum of the rates of pressure rise due to residual and induced gas in-leakage, less the rate of pressure drop due to cryopumping. The residual in-leakage is composed of the in-leakage of air through the gaskets, seals, etc., plus the load from the outgassing of materials within the system. Outgassing is an evolution of gas from a material and occurs when the pressure of the system approaches the vapor pressure of the material. The induced in-leakage is a metered amount of gas introduced into the chamber to evaluate the pumping performance of the cryopanel. Thus

$$\dot{P} = \frac{P_r \dot{V}_r}{V} + \frac{P_i \dot{V}_i}{V} - \frac{P \delta A}{V} \left(\frac{R_o T}{2 \pi M} \right)^{1/2} \qquad (7)$$

where the subscript, r , indicates residual in-leakage and subscript, i , indicates induced in-leakage, and P is the pressure within the chamber.

Now without cryopumping and induced in-leakage, (7) reduces to

$$\dot{P} = \frac{P_r \dot{V}_r}{V} \quad \text{or} \quad \dot{P} V = P_r \dot{V}_r \tag{8}$$

If the outgassing rate is considered as a virtual in-leakage of air, it may be combined with the real in-leakage rate of air, and the residual in-leakage volume rate, $\dot{V}_r$, may be determined. By pumping the system down to the desired pressure with the mechanical and diffusion pumps, closing the valves to the pumps, and observing the initial rate of change of pressure, $\dot{V}_r$, the residual volume in-leakage rate (at pressure P_r) may be determined directly from (8).

The induced in-leakage volume rate is determined by knowing the conditions of the induced gas (P_i and T_i) and the induced mass rate of flow.

Now with cryopumping, (7) applies, and at equilibrium the rate of change of pressure is zero and

$$\frac{P_r \dot{V}_r}{V} + \frac{P_i \dot{V}_i}{V} = \frac{P \delta A}{V} \left(\frac{R_o T}{2\pi M} \right)^{1/2} \tag{9}$$

and multiplying by the chamber volume, V ,

$$P_r \dot{V}_r + P_i \dot{V}_i = P \delta A \left(\frac{R_o T}{2\pi M} \right)^{1/2} \tag{10}$$

where P is the equilibrium pressure obtained by cryopumping.

As the pressure is lowered and approaches the vapor pressure of the condensed phase, the net number of molecules pumped is equal to the number sticking to the plate minus the number leaving the plate due to evaporation, and from (10):

$$P_r \dot{V}_r + P_i \dot{V}_i = \delta A \left(\frac{R_o}{2\pi M} \right)^{1/2} \left[P T^{1/2} - P_c T_c^{1/2} \right] \tag{11}$$

where $P_r \dot{V}_r$ may be obtained by the use of (8), $P_i \dot{V}_i$ is measured by knowing the conditions of the induced gas, A is the panel area, R_o is the universal gas constant, M is the molecular weight of the induced gas, P is the chamber pressure, T is the temperature of the gas in the chamber, P_c is the equilibrium vapor pressure of the condensed gas on the cryopanel at temperature T_c and may be obtained from App. VIII. Thus the capture coefficient, δ, is the only unknown and may be determined from (10), or if the condensate vapor pressure is significant from (11).

We define the system throughput, Q, as equal to the in-leakage rate at equilibrium, thus

$$Q = P_r \dot{V}_r + P_i \dot{V}_i \tag{12}$$

Then the pumping speed, S, at pressure, P,

$$S = Q/P \tag{13}$$

The cryopanel pumping speed, S', is defined as the volume flow rate, at chamber pressure, condensed per unit area of the cryosurface. This may be determined by obtaining the total speed of the cryopanel plus the diffusion pump and subtracting the speed of the diffusion pump, then dividing by the panel area:

$$S' = \frac{1}{A}(S_t - S_d) \tag{14}$$

The diffusion pump speed may be found by attaining the ultimate pressure, Po, without cryopumping and without induced in-leakage. If the chamber is then isolated from the diffusion pumping system and the initial rate of change of chamber pressure with time is recorded, $\frac{dP_o}{dt}$, the

diffusion pump speed may be found, thus

$$S_d = \frac{\frac{dP_o}{dt} V}{P_o} = \frac{Q_o}{P_o} \qquad (15)$$

The diffusion pump speed is nearly constant below 10^{-4} torr. and will be considered such in the following calculations, as normally the chamber will be operated at pressures below this value.

The total pumping speed, St, is found by observing the chamber pressure, P, for a known in-leakage rate $P_i \dot{V}_i$, obtained by cryopumping. The total pumping speed is now

$$S_t = \frac{P_r \dot{V}_r + P_i \dot{V}_i}{P} = \frac{Q}{P} \qquad (16)$$

The cryopanel pumping speed, S', may now be found from (14).

Let us now compute, for our system, the theoretical minimum pressure that the cryopump can reach as a function of the throughput. The maximum pumping will occur when the capture coefficient, δ, is unity. As the vapor pressure of solid nitrogen at 20°K is about 10^{-10} torr. (App. VIII), and may be neglected at pressures much greater than this, eq. (10) may be used with (12) to give:

$$Q_p = P \delta A \left(\frac{R_o T}{2\pi M}\right)^{1/2} \qquad (17)$$

where $Q_p = Q_{total} - Q_{diff.pump}$

$$\therefore \quad P_{min} = \frac{Q_p}{\delta A} \left(\frac{R T}{2\pi M}\right)^{-1/2} \qquad (18)$$

System Parameters:

$A = 1 ft^2 = 929 cm^2$

$Ro = 8.314 \times 10^7$ erg/°C-gm mole

$$1 \text{ erg} = \frac{1 \text{ gm} - \text{cm}^2}{\text{sec}^2 - °\text{K-gm mole}}$$

$t = 300°K$

M = 28 gms/gm mole for N_2 gas

$Q = \mu$-l/sec $= \frac{\text{mm Hg-cc}}{\text{sec}}$

$\delta = 1$

Substitution into (18) gives:

$$P_{min} = 9.05 \times 10^{-8} Q \text{ mmHg} \tag{19}$$

and

$$Q \frac{\mu\text{-l}}{\text{sec}} = (760 \text{ mmHg})\left(\frac{1000\mu}{\text{mmHg}}\right)\left(\frac{1\,\ell}{1000\,\text{cc}}\right)\left(\dot{V}\,\frac{\text{cc}}{\text{sec}}\right)_{stp} \tag{20}$$

thus

$$\dot{V}_{stp} = 1.315 \times 10^{-3} Q \frac{\text{cc}}{\text{sec}} \tag{21}$$

Also,

$$S'_{max} = \frac{Q_p}{A\,P} \frac{\ell}{\text{cm}^2\text{-sec}} \tag{22}$$

By the use of (19), (21), (22) and assumed values of Q, the following table is obtained.

TABLE I

Qp 1/sec	Pmin-mm Hg	stp-cc/sec	S'max-1/sec-cm^2
0.1	9.05×10^{-9}	1.315×10^{-4}	11.9
1.0	9.05×10^{-8}	1.315×10^{-3}	11.9
10.	9.05×10^{-7}	1.315×10^{-2}	11.9
100.	9.05×10^{-6}	1.315×10^{-1}	11.9
1000.	9.05×10^{-5}	1.315	11.9

This table is illustrated in the accompanying graph.

Barrington, /23/, among others /8, 15/, uses the concept of conductance

to obtain the pumping speed of the cryopump. If the assumption is made that the vapor pressure of the gas condensate is much less than the chamber pressure, the total speed of the cryopump for a gas at 25°C as determined by the conductance concept is:

$$S = \frac{405A}{\sqrt{M}} \frac{\ell}{sec} \quad (A \text{ in sq. in.}) \tag{23}$$

For the present system with a panel area of 144 in^2 and pumping nitrogen gas with molecular weight of 28 gm/mole, (23) gives:

$$S = \frac{405(144)}{\sqrt{28}} = 11{,}020 \ \ell/sec \tag{24}$$

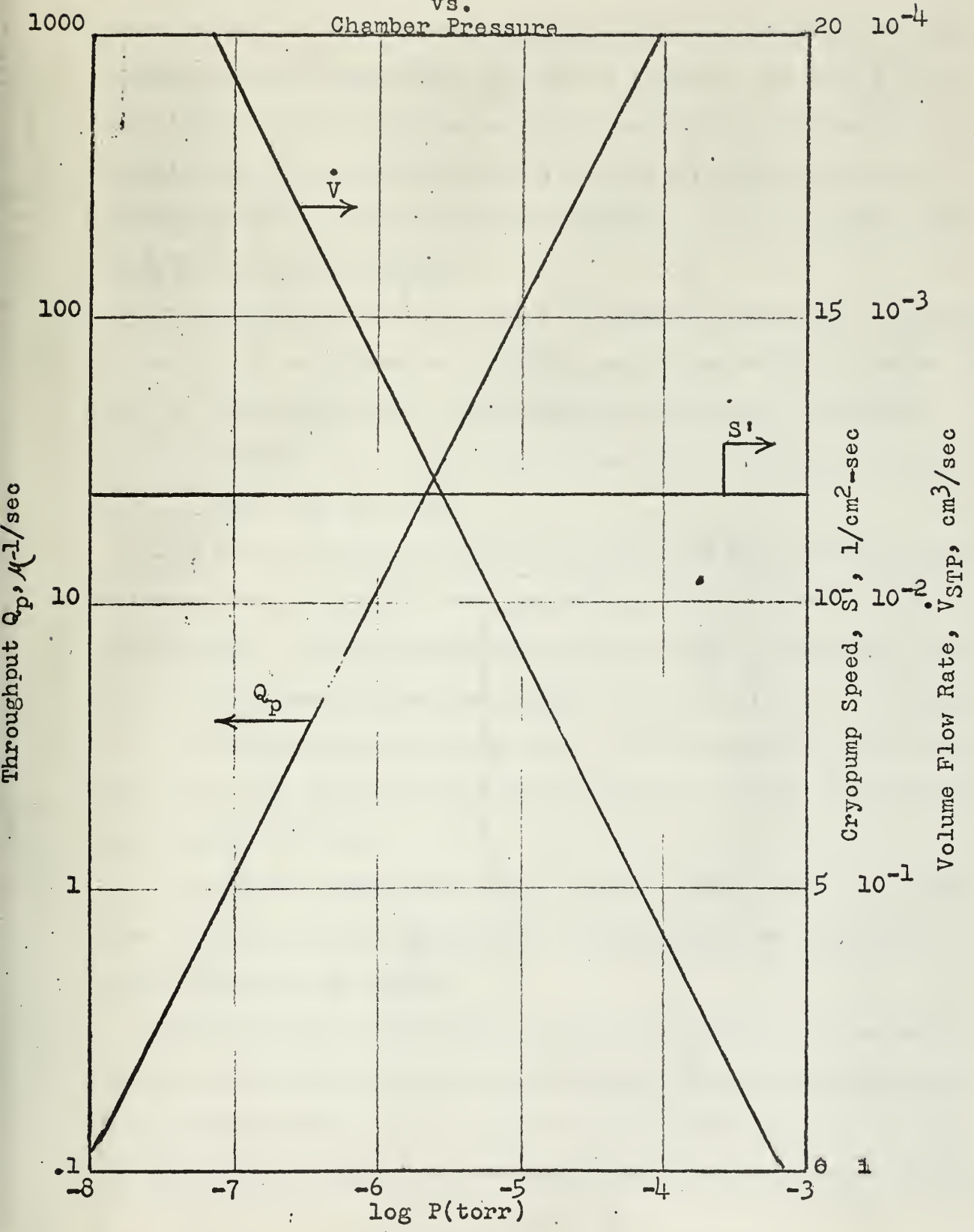
dividing by the panel area of 928 cm^2

$$S' = \frac{11020}{928} = 11.9 \ \ell/sec\text{-}cm^2 \tag{25}$$

Thus if the same assumptions are made, the results of the theory as developed in this thesis are in agreement with the results as determined from the conductance approach.

GRAPH I

Throughput, Cryopump Speed
and Volume Flow Rate
vs.
Chamber Pressure



3. Construction of Chamber and Cryopump.

The basic components for the vacuum chamber were obtained by modifying a standard 40 inch diameter vacuum induction furnace manufactured by National Research Corporation. The heating elements, crucible, and all associated components were removed and all unnecessary openings were blanked with stainless steel plates and sealed by welding with a low outgassing welding material, "Eutec-StainTrode A", which is manufactured by Eutetic Welding Alloys Corp.

The roughing pump, used to reduce the chamber pressure from atmospheric pressure to a few microns, is a 100 CFM, single-stage mechanical vacuum pump and the holding pump is a 1.25 CFM mechanical vacuum pump. Blank-off pressure of these pumps is 10 to 15 microns. For the schematic of the pumping system, see App. I(a).

The booster diffusion pump of the original NRC system, which had an ultimate pressure of 1×10^{-4} torr., was replaced with a 6 inch, 4 stage, fractionating, diffusion pump with an ultimate blank-off pressure of 10^{-7} to 10^{-9} torr. depending upon the pumping fluid used. (1 torr. = 1 mm Hg).

The diffusion pump was charged with a silicone diffusion pump fluid, Dow Corning 704, which gives the diffusion pump an ultimate blank-off pressure of about 10^{-7} torr.

Most of the original cast iron piping and flanges were replaced with Type 304 stainless steel piping and flanges to reduce the problem of outgassing within the system.

Where possible, particularly on the high vacuum side, all gasketed fittings were replaced with O-ringed fittings. Where space allowed double O-ring fittings were used. The outer O-ring was made of butyl, which has low gaseous permeability, and the inner O-ring of silicone rubber, which has

low outgassing properties. On fittings where there was room for only one O-ring, butyl was used.

Cryopumps studied by previous investigators have recirculated the refrigerant in a closed loop through the cryopanel then to a cryostat for recondensation /15/. In this thesis, a "batch process" cryopump was constructed, that is, a charge of liquid helium is introduced into the panel. As the helium absorbs heat and vaporizes, it is vented to the atmosphere. As the panel begins to warm, a new charge of liquid helium is introduced.

The cryopanel designed and installed for the initial cryopumping studies is a one foot square, copper plate, backed by a network of thin-wall, copper tubing which is to be filled with liquid helium (See Figure 4(a) and 4(c)). This configuration provides a panel with a minimum of mass to be cooled, thus requiring a minimum of liquid helium. Copper was chosen as the panel material because of its high thermal conductivity, thus it tends to maintain a uniform temperature distribution. The boiling temperature of liquid helium at atmospheric pressure is 4.2°K, thus the panel should be at a temperature below 20°K.

Two other cryopanels are available for future testing. Both of these are 12"x18" panels of Type 304 stainless steel. One is single embossed and the other has embossing on both sides.

At the low operating temperatures of the cryopanel, the radiation heat load to the cryopanel from the vacuum chamber walls at nearly room temperature would be considerable. At the low chamber pressures (below 10^{-4} torr.) the heat transfer to the panel by gas conduction is negligible when compared to the radiation load. Thus, to minimize the refrigeration requirement of the panel, it is necessary to provide a radiation shield between

the cryopanel and the chamber walls.

The radiation shield, Fig. 4, consists of a 33 inch diameter, 36 inch long, Type 304 stainless steel, single embossed, cylindrical shell and 36 inch diameter, single embossed, Type 304 stainless steel end shields. The radiation shield and the two stainless steel cryopanels were manufactured by Dean Products, Inc., of Brooklyn, N. Y., using their standard "Panelcoil" embossing. The cylinder and end panels are cooled with liquid nitrogen to about 77°K thus reducing the radiation heat load on the cryopanel to about .43% of the load without shielding /App. VI/.

In a similar manner, the radiation load on the liquid nitrogen heat shield may be reduced by the use of another shield between it and the chamber wall. Using a dry, aluminum shield for this purpose, with the more reflective side toward the chamber walls, the radiation heat load on the nitrogen filled shield can be reduced to about 38% of its unshielded value /App.VI/. In this system, it was possible to dry shield only about 75% of the nitrogen filled shield in order to leave instrumentation ports open and to facilitate removal of the liquid nitrogen filled shield. However, this provides better than 50% reduction of the heat load on the primary heat shield.

In areas where there is contact between the cold heat shield and the chamber wall, such as the supporting legs of the shield, Teflon is used to act as a thermal barrier. It has low thermal conductivity (.11 BTU/hr.-ft^2-°F) and low outgassing properties.

The liquid nitrogen filled heat shield has a volumetric capacity of about 33 liters. When filling the shield, boiloff of the liquid nitrogen occurs until the shield has cooled down. To accommodate the boiloff and to fill the heat shield requires a total of approximately 65 liters of

liquid nitrogen. The copper cryopanel has a capacity of .4 liters and requires 20 to 25 liters of liquid helium to cool it down and fill it. This total may be reduced about 50% by precooling the panel with liquid nitrogen prior to filling it with liquid helium.

The stainless steel piping for the heat shield and the cryopanel undergo changes in temperature of about 220°C and 290°C respectively when the coolants are introduced. This causes considerable contraction of the tubing, and to accommodate this contraction, there is a section of "Keflex" 3/4 inch stainless steel flexible tubing in each section of the line. /Fig. 4/.

The cylindrical shield and the rear end panel are piped together with common fill and common vent lines. The front end shield is mounted in the door and moves with it to permit access to the cryopanel. /Figs. 4 & 5/.

The heat shield and the cryopanel are both piped so that the liquid is introduced from the bottom and vented at the top. /Fig. 4(b)/. This helps to eliminate gas pockets and surging caused by the boiling off of the liquid. The cryopanel fill and vent lines and the common fill and vent lines from the cylindrical end shield and the back panel are located in the flanged opening at the top of the chamber. The fill and vent lines for the end panel are mounted in the door, protrude through the top of the door and are vacuum sealed by a soft soldered joint.

An electrical feed-through plate is installed on one of the flanged openings on the side of the chamber. In it are mounted three, six-pin hermetic feed-throughs and three, eight-pin hermetic feed-throughs, thus giving a capability of monitoring 21 two-lead elements (e.g. thermistors and thermocouples) within the system. Also provided on this plate are

openings for a thermocouple gage and McLeod gage. These may be sealed off if these gages are not in use.

4. Instrumentation.

a) Pressure.

Pressures above one micron (10^{-3} torr.) are monitored by the use of thermocouple gages, Type DV-1M, manufactured by Vacuum Electronics Co. The location of these gages is shown on the schematic diagram of the system/App. I/. The thermocouple gage controller was calibrated against a McLeod gage, and all gages agreed within about five microns of each other.

Pressures below one micron are monitored by a Bayard-Alpert type ionization gage. This type of gage has a range of 10^{-3} to 10^{-9} torr. and is accurate within two percent. As this gage is mounted outside the heat shield, it acts as a non-directional gage and senses average pressure.

b) Surface Temperatures.

The surface temperatures of the liquid nitrogen filled heat shield were monitored by the use of disk thermistors, Type L0904-125K-H-T2, manufactured by Keystone Carbon Co. They were individually calibrated by the manufacturer at 90°K, 77.1 °K, and 20.2°K. A typical calibration curve for the liquid nitrogen range thermistors is shown in Appendix V. As the resistance of these thermistors is of the order of magnitude of 500 to 800 ohms at the temperature of liquid nitrogen and the total lead and contact resistance is about one ohm, the lead and contact resistance may be considered negligible.

The thermistor locations on the heat shield cylinder are midway between the ends, at about 45° above and below the horizontal centerline on the inner surface. On the end shields the thermistors are located on the vertical centerline at about the 1/3 and 2/3 diameter points.

One copper-constantan thermocouple is mounted next to a thermistor on

the cylindrical heat shield to compare the temperature as indicated by the thermistors with that indicated by the thermocouple.

The surface temperatures of the cryopanel are obtained by the use of thermistors of type LO904- 3meg-He-T2. These were individually calibrated by the manufacturer at 90°K, 77.1°K, 20.2°K, and 4.1°K. A typical calibration curve for this type of thermistor is shown in Appendix V.

Three thermistors are located on the cryopanel; one at the top and at the bottom on the vertical centerline, and one at a top corner. This gives a twofold result. First, it provides a method of measurement of the liquid level within the panel. Secondly, it is a means of determining if there is a temperature distribution across the panel, as the coldest point on the panel is at the lower center, and if there is a distribution, the warmest spot would be expected at a top corner of the panel.

The thermistors are bonded to the surface with SAUEREISEN TYPE 29 "LOW EXPANSION CEMENT". This cement gives a combination of good heat conduction and good electrical insulation. Resistance to ground for all cemented units was found to be nearly infinite. A drawback of this type of cement is that it is porous, and thus outgases until the entrapped gas can be drawn out of the pores.

Glass insulated copper leads are used for the thermistors. These also outgas, but not as severely as the cement.

The resistance of the thermistors is measured by the use of a wheatstone bridge. All the thermistors are fed into a selector switch and then to the bridge; thus rapid readings may be obtained.

c) In-leakage Flow Rates.

The induced leak is obtained by admitting gaseous nitrogen into the chamber. A schematic of the gas admission system is shown in

Appendix I(b). The system is sealed from the chamber by a high vacuum valve located in the center of the door. When gas is to be admitted, this valve is opened and the gas flow rate is controlled by a needle valve. The gas flow rates are monitored by the use of a rotometer. As flow rates are widely varied, several different rotometers must be used. A typical rotometer calibration curve is shown in Appendix IV. This curve was obtained by calibrating the rotometer against a positive displacement gas flow meter.

5. System Checkout Procedure.

a) Establishing the initial vacuum prior to cryopumping.

The mechanical pumping system was isolated and the blank-off pressure of the mechanical pumps was determined to be 10 to 15 microns for each pump. This pressure was measured by thermocouple gages located in the foreline of each pump.

The vacuum chamber was then evacuated with the roughing pump. The ultimate chamber vacuum attainable with the roughing pump was 15 to 20 microns. Upon each evacuation by the roughing pump, gas ballasting was used for about 15 to 20 minutes. This procedure admits air on the compression stroke and aids in the removal of condensables from the system.

After the system had reached the lowest pressure attainable with the roughing pump ($\sim 20\mu$), the diffusion pump was placed on the line. (For procedures, see App. II.) The time required to heat up the diffusion pump prior to placing it on the line requires about 20 minutes.

Initially, with the diffusion pump on the line, the lowest pressure attainable was 8×10^{-4} torr. The system was then checked with a Veeco mass spectrometer type leak detector using helium as the probing gas. Several minor leaks in joints and flanges were found and corrected. Then it was found that in areas where heat had been applied, such as seal welds, the walls of the chamber had become porous. To remedy this, the affected areas were painted with GLYPTAL, a sealant paint. This was done with vacuum in the chamber so that the sealant was drawn into the pores. Upon drying, the pores were effectively sealed. Many of these porous spots were found and to completely eliminate this problem the entire exterior of the chamber was finally painted with GLYPTAL. After this was accomplished and all detectable leaks had been corrected, the lowest vacuum attainable was 1×10^{-5} torr.

This was the best vacuum attainable without cryopumping and it generally required 12 to 24 hours of pumping to reach this level due to the outgassing load within the chamber. This outgassing load is believed to consist primarily of the cement used to bond the thermistors and thermocouples, and the insulation on their lead wires.

b) Checking the liquid coolant systems.

The liquid coolant systems were checked by filling them with liquid air. This was done to insure that the joints in the lines and the vacuum seals would withstand the thermal shocks and the very low temperatures involved.

The liquid filled heat shield and the cryopanel were both filled with liquid air and all the lines held up well. Filling was initially accomplished by pouring directly from 50 liter liquified gas containers (dewars) into a funnel in the filling lines. The cryopanel and door mounted end shield filled easily and no trouble was encountered. On filling the cylindrical heat shield however, geysering began to occur when it was about 2/3 full. As this presented a personnel safety hazard, another method of filling was attempted.

A transfer tube was inserted in the dewar and the liquid air was pumped directly into the system by subjecting the dewar to an internal pressure of a few psig of gaseous helium. This method, which is a common one for transferring cryogenic fluids, proved easier and required less liquid air, as less was lost due to boil-off in the funnel. This same method may be employed for transferring the liquid helium from its dewar into the cryopanel. The geysering hazard was eliminated by the use of baffled surge cylinders over the fill and vent lines. This allowed the vapor to escape without carrying the liquid coolant with it. To completely

eliminate the problem, it is felt that the capacity of the venting system for the cylindrical heat shield must be enlarged.

c) Checking the Thermistors.

With the coolant system filled with liquid air, the surface temperature of the heat shield as measured by the thermistors was compared with the temperature as measured by a copper-constantan thermocouple. The temperatures as measured by the thermistors agreed within 10° of the temperature as measured by the thermocouple (93°K). All the thermistors read lower than the thermocouple. It is to be noted that the sensitivity of the thermocouple at this temperature is 18.04 μV/°K while the sensitivity of the thermistors is about 15 Ω/°K, depending on the thermistor used. Using the wheatstone bridge, the thermistor resistance could be accurately read to one ohm. As the temperatures obtained by the thermistors did not correlate well, it is felt that they can be used to give a good indication of the liquid coolant level, but the temperature that is observed is only approximate. Thus it is more advantageous to use copper-constantan thermocouples on the heat shield.

On the cryopanel, where the temperature is below 20°K, the sensitivity of the thermocouples is so small (less than 5.0 μV/°K) that they are impractical and thermistors must be used. With the liquid helium range thermistors, the sensitivity is about 35 Ω/°K and thus greater accuracy should be obtained than with the liquid nitrogen range thermistors.

6. Experimental Procedure.

(1) Pump the vacuum chamber to as low a pressure as possible using the mechanical and diffusion pumps.

(2) Fill the heat shield with the liquid coolant. Filling time is about 3-1/2 hours. About 65 liters of coolant are required.

(3) Record all heat shield temperatures and ultimate chamber pressure obtainable without cryopumping, P_o.

(4) Isolate the chamber from the diffusion pump and record the initial rate of change of chamber pressure, dP_o/dt.

(5) Diffusion pump speed may then be found.

$$S_d = Q_o/P_o = \frac{V\frac{dP_o}{dt}}{P_o}$$

This speed may be assumed constant at all chamber pressures below 1×10^{-4} torr.

(6) Admit the pumps to the vacuum chamber and restore ultimate chamber pressure, P_o.

(7) Precool the cryopanel by filling it with liquid nitrogen.

(8) Purge the liquid nitrogen from the cryopanel by introducing gaseous helium.

(9) Fill the cryopanel with liquid helium. Rapid boiloff of the liquid helium may require that the filling process be continuous in order to supply the necessary makeup.

(10) Record temperatures of the panel and heat shield and the chamber pressure, P, obtained by cryopumping.

(11) Total pumping speed is then

$$S_t = Q_o/P_o$$

(12) Cryopanel pumping speed at pressure P may now be found

$$S' = \frac{S_t - S_d}{A}$$

(13) Induce N_2 gas to system, recording P_i and $\dot{V}_i$.

(14) Throughput now is

$$Q = Q_o + P_i \dot{V}_i$$

(15) Record cryopanel and heat shield temperatures and chamber pressure, P_1.

(16) Total pumping speed is

$$S_{t_1} = Q/P_1$$

(17) Cryopanel pumping speed at pressure P_1 is

$$S_1' = \frac{S_{t_1} - S_d}{A}$$

(18) Repeat steps (13) thru (17), varying the product $P_i \dot{V}_i$ for each run.

(19) Cryopump performance curve of S' vs. P may now be plotted.

(20) Capture coefficient, γ , at each pressure maybe determined from

$$\gamma = \frac{Q}{A}\left(\frac{2\pi M}{R_o}\right)^{1/2}\left[P T^{1/2} - P_c T_c^{1/2}\right]^{-1}$$

7. Conclusion.

a) A system for cryopump evaluation was constructed and instrumented.

b) A preliminary check-out was performed to insure that the system would maintain vacuum and that it would withstand the thermal shocks caused by the liquid coolants and the low operating temperatures.

c) A theoretical method of evaluating the cryopump performance was presented. The theoretical pumping speed at chamber pressures greater than 10^{-10} torr. is predicted to be constant at about 10 l/cm^2-sec. For an in-leakage of .1 μ-l/sec, which is considered to be the minimum practicably obtainable, the ultimate chamber pressure is predicted to be about 10^{-8} torr. This corresponds to an altitude of about 520 kilometers (323 miles), thus the chamber may be used as a space simulation chamber.

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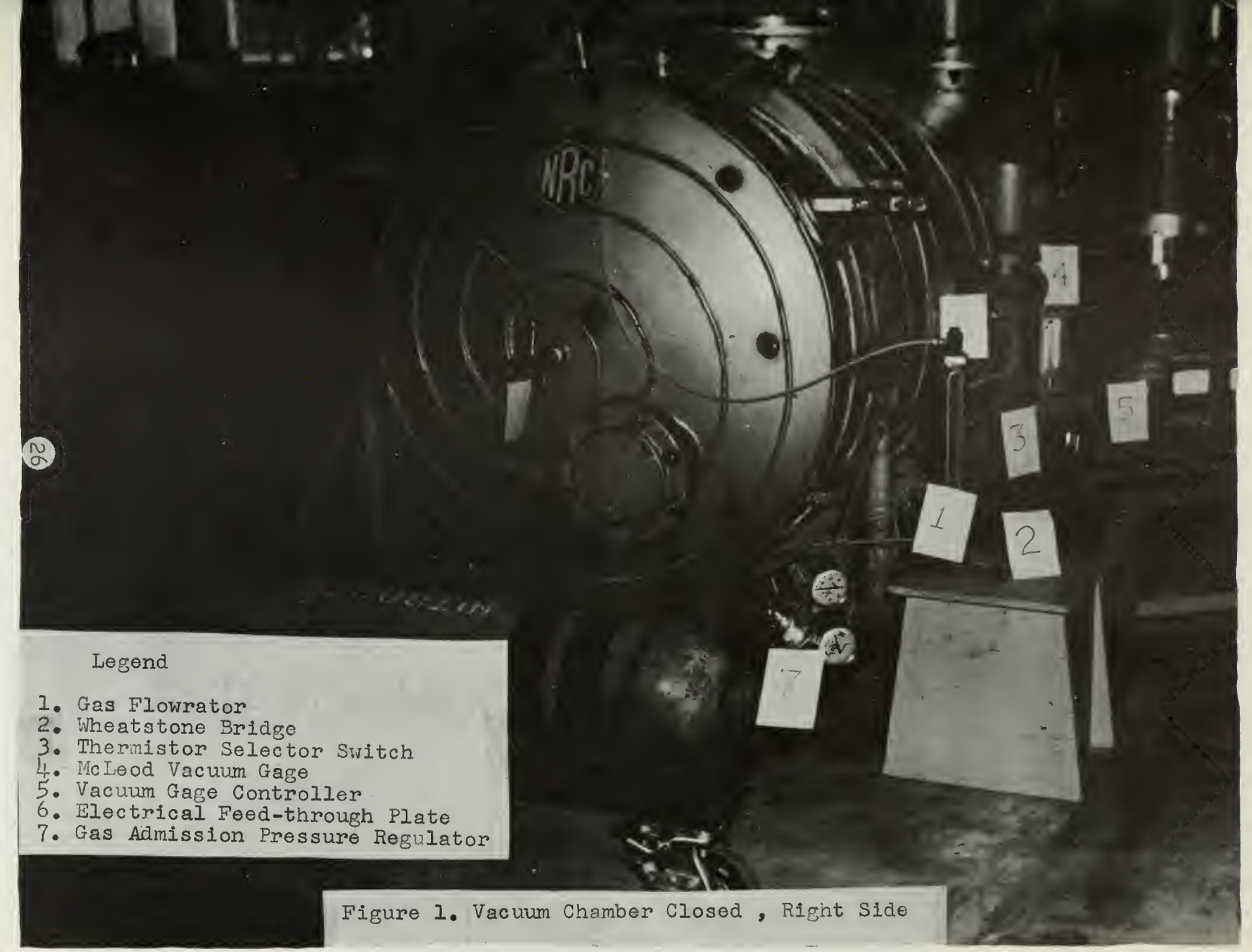
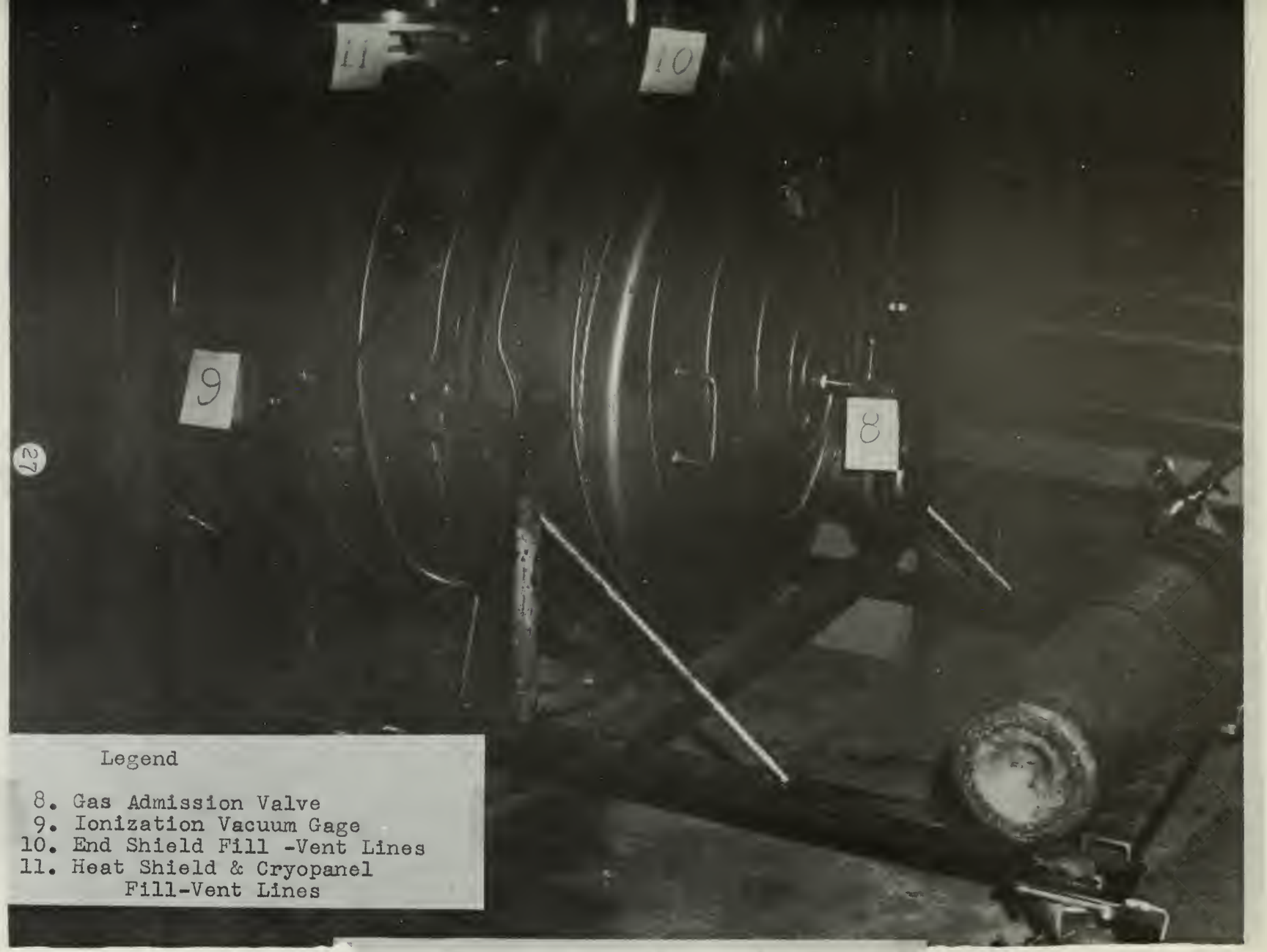


Legend

1. Gas Flowrator
2. Wheatstone Bridge
3. Thermistor Selector Switch
4. McLeod Vacuum Gage
5. Vacuum Gage Controller
6. Electrical Feed-through Plate
7. Gas Admission Pressure Regulator

Figure 1. Vacuum Chamber Closed , Right Side



Legend

8. Gas Admission Valve
9. Ionization Vacuum Gage
10. End Shield Fill -Vent Lines
11. Heat Shield & Cryopanel Fill-Vent Lines

Figure 2. Vacuum Chamber Closed , Left Side

Figure 3. Vacuum Chamber Open , Showing Dry Heat Shield

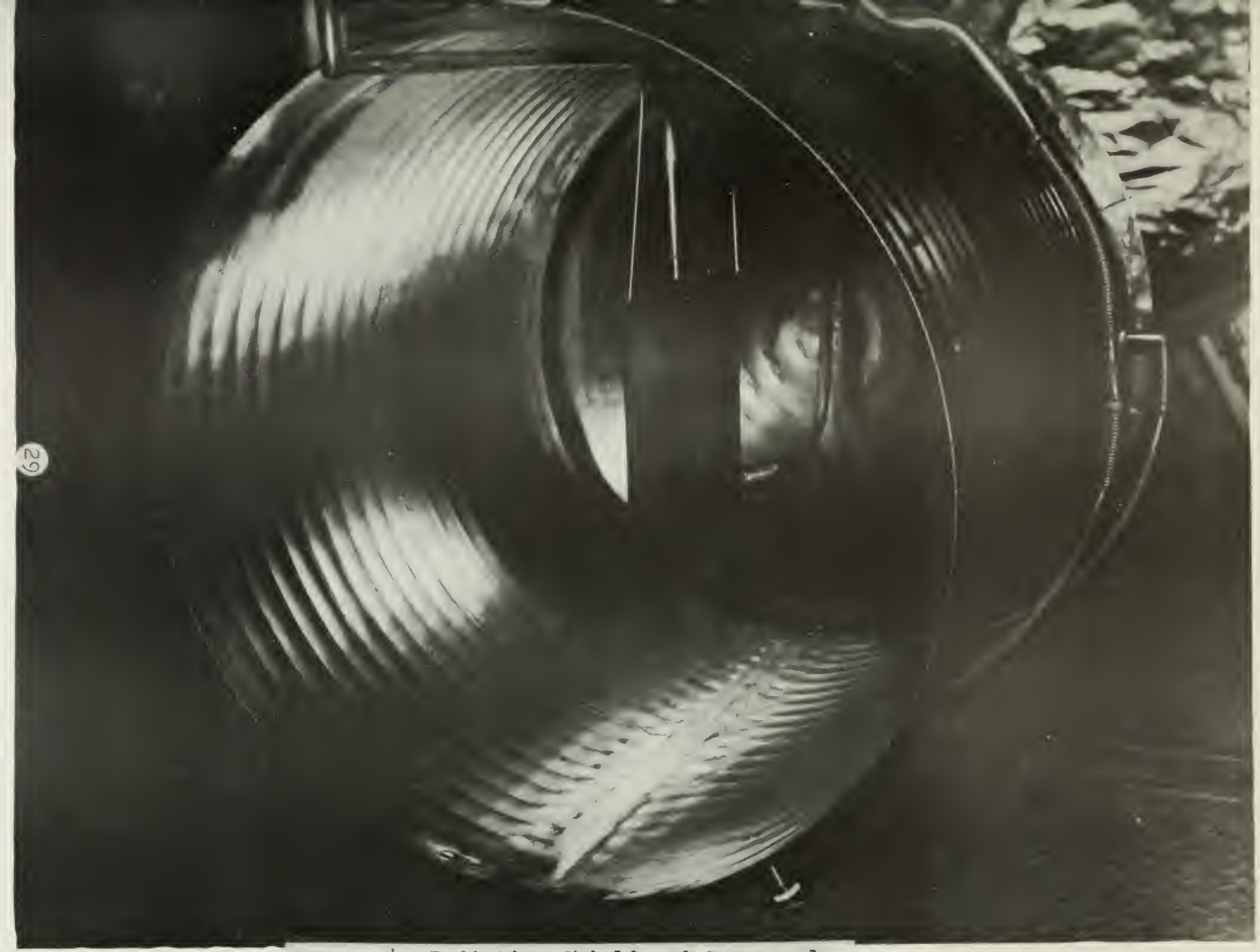


Figure 4. Radiation Shield and Cryopanel

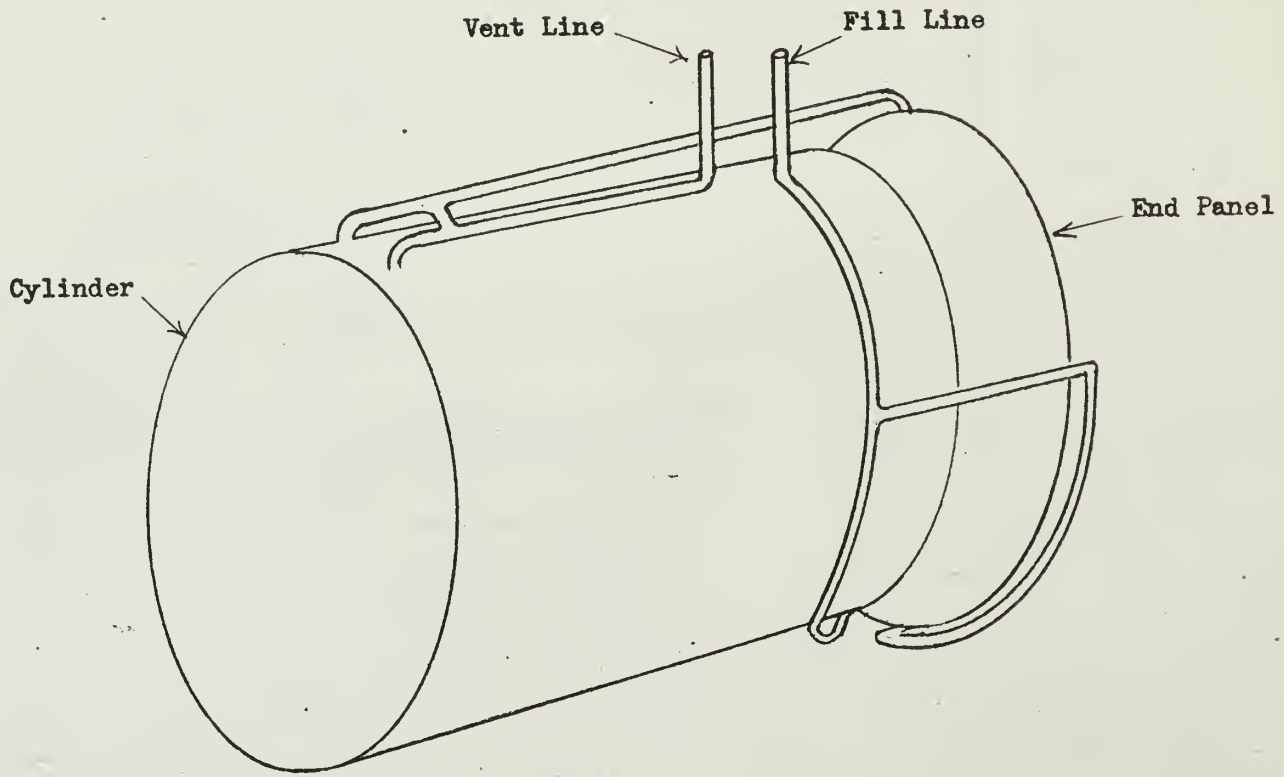


Figure 4(b) Sketch of Heat Shield Piping

1. 12"x12"x$\frac{1}{16}$" Copper Plate

2. .75" O.D. Thinwall Copper Tube

3. .375 O.D. Thinwall Copper Tube

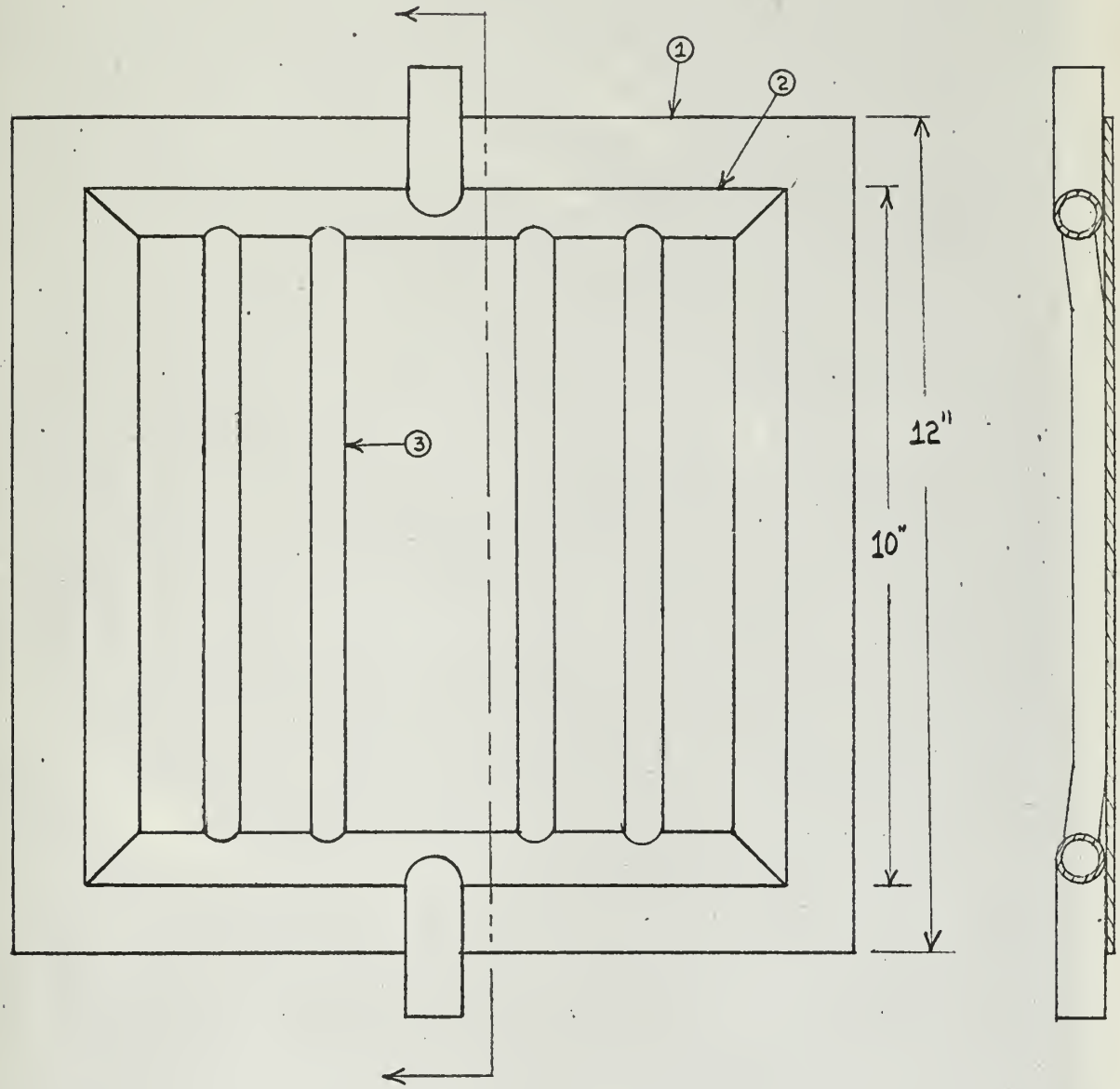


Figure 4(c) Sketch of Cryopanel

[illegible] and [illegible]orward [illegible]nd [illegible]anel

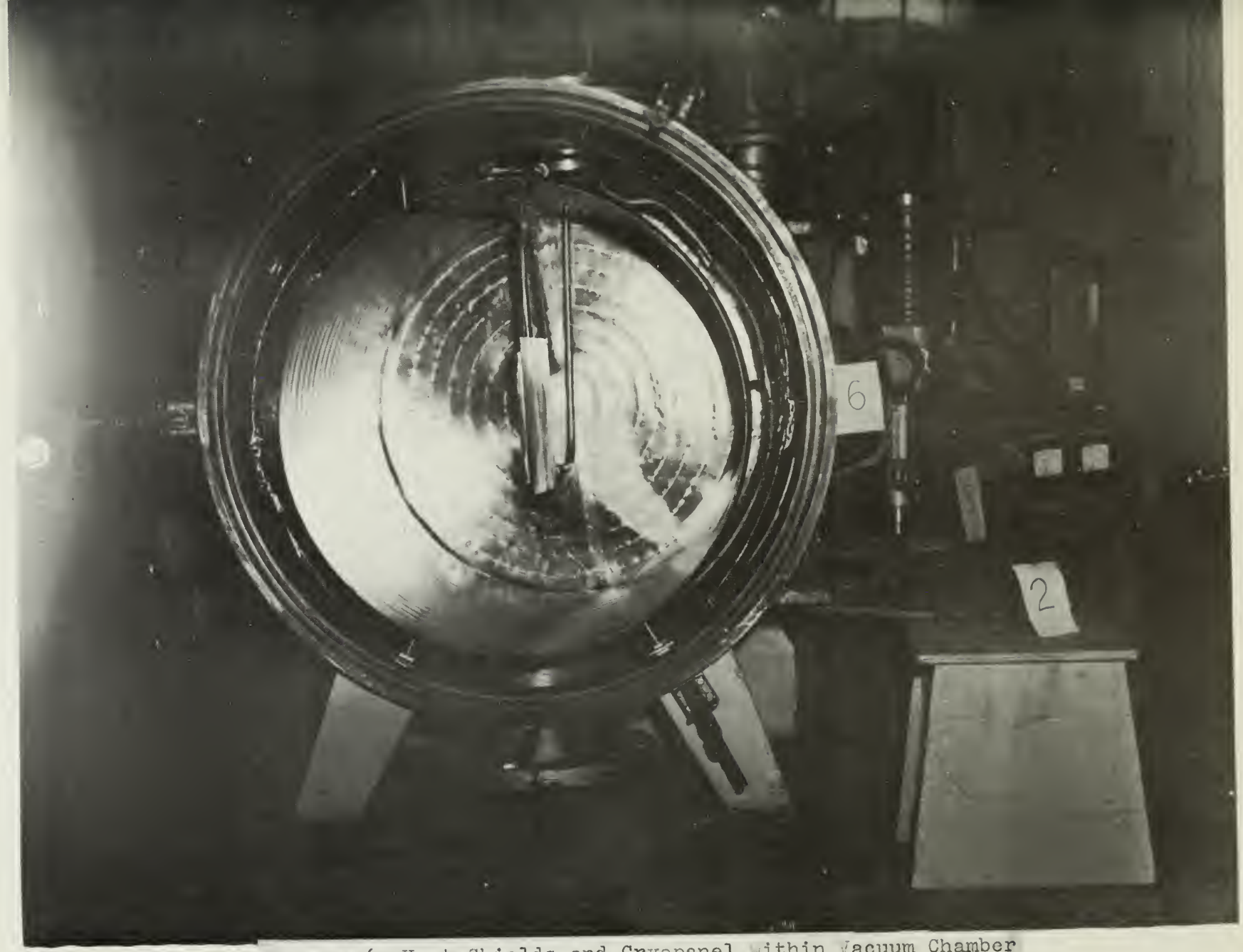
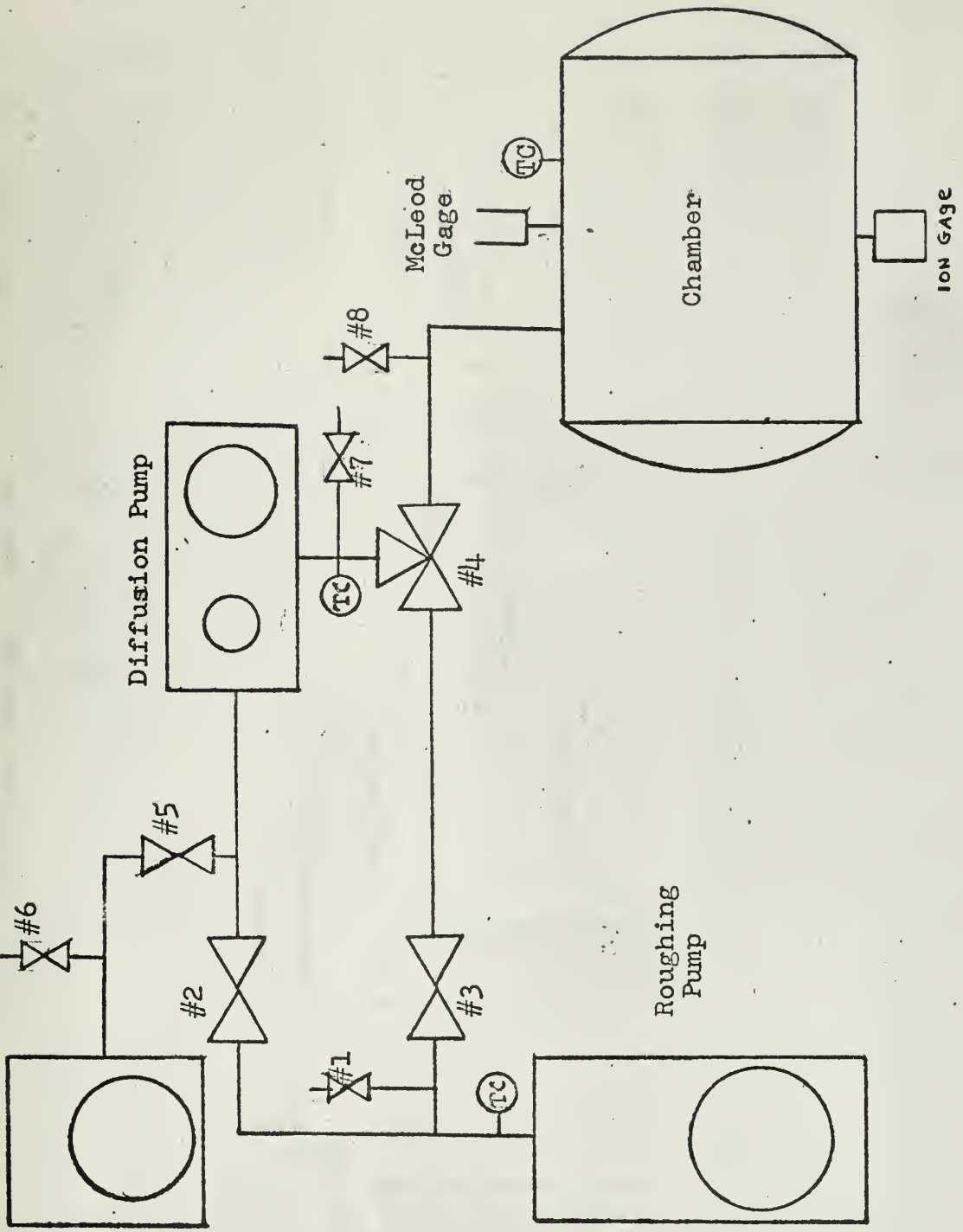


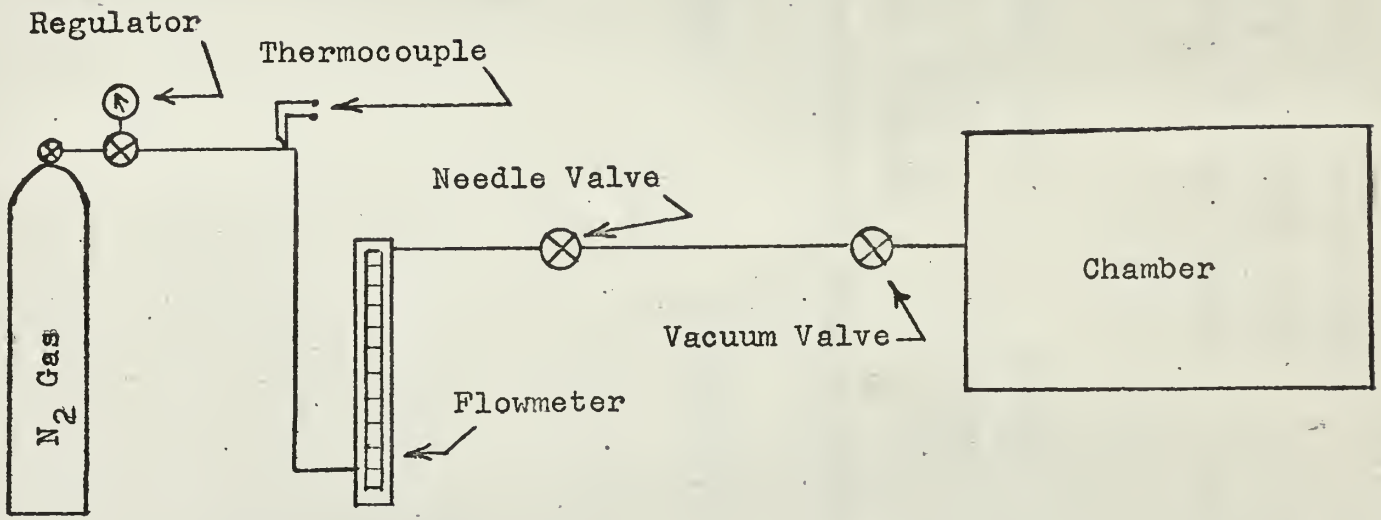
Figure 6. Heat Shields and Cryopanel Within Vacuum Chamber

APPENDIX I

I(a) Pumping System Schematic



I(b) Gas Admission System



I(c) - System Dimensions

A. Length:

Chamber diameter	41 in. = 104 cm.
Chamber length	46 in. = 116.6 cm.
Cylindrical Shield Diameter	33 in. = 83.7 cm.
Cylindrical Shield Length	36 in. = 91.3 cm.
Shield End Panel Diameter	36 in. = 91.3 cm.

B. Area:

Chamber Walls	8.56×10^{-3} in^2 = 5.51×10^{-4} cm^2
Heat Shield*	5.44×10^{-3} in^2 = 3.51×10^{-4} cm^2
Copper Cryopanel*	144 in^2 = 928 cm^2

*Projected surface area, does not include embossing.

C. Volume:

Chamber	6.06×10^{4} in^3 = 994 liters

APPENDIX II

PUMPING SYSTEM OPERATING PROCEDURES

To evacuate the chamber:

A. Insure that all doors, ports, and air release valves #1, 6, 7 & 8 are closed.

B. Close valve #4 on top of the diffusion pump.

C. Open valve #5 to the mechanical holding pump.

D. Open valve #3 in the roughing line.

E. Close valve #2 in the diffusion pump foreline.

F. Start the mechanical roughing pump, thus evacuating the tank.

G. Start the mechanical holding pump, thus evacuating the diffusion pump.

H. Wait until the pressure in the tank reaches approximately 70 microns or lower. The pressure in the diffusion pump should also be approximately 70 microns or lower.

I. Start the diffusion pump. Insure that the cooling water is on.

J. Open valve #2 between the diffusion pump and the mechanical roughing pump.

K. Close valve #5 to the mechanical holding pump.

L. Open valve #4 on top of the diffusion pump.

M. Close valve #3 in the roughing line.

N. The gas flow is now from the tank, through the diffusion pump, to the mechanical roughing pump.

If it is necessary to open the tank:

A. Close valve #4 on top of the diffusion pump.

B. Open air release valve #8.

C. Leave pumps running.

The tank may now be opened.

To re-evacuate the tank:

A. Open valve #5 to the mechanical holding pump.

B. Close valve #2 in the diffusion pump foreline.

C. Close air release valve #8 and insure that all ports are closed.

D. Open valve #3 in the roughing line.

E. Wait until the pressure in the tank is 70 microns or less.

F. Open valve #2 between the diffusion pump and the roughing pump.

G. Close valve #5 to the holding pump.

H. Open valve #4 on top of the diffusion pump.

I. Close valve #3 in the roughing line.

To shut the pumping system down:

A. Close valve #4 on top of diffusion pump.

B. Turn off the diffusion pump and turn on the quick cool water.

C. Wait approximately 15 minutes for the diffusion pump to cool.

D. Close valve #2. Admit air to the diffusion pump and tank by opening valves # 7 & 8.

E. Turn off both mechanical pumps and open air release valves # 1 & 6.

F. System is now secured.

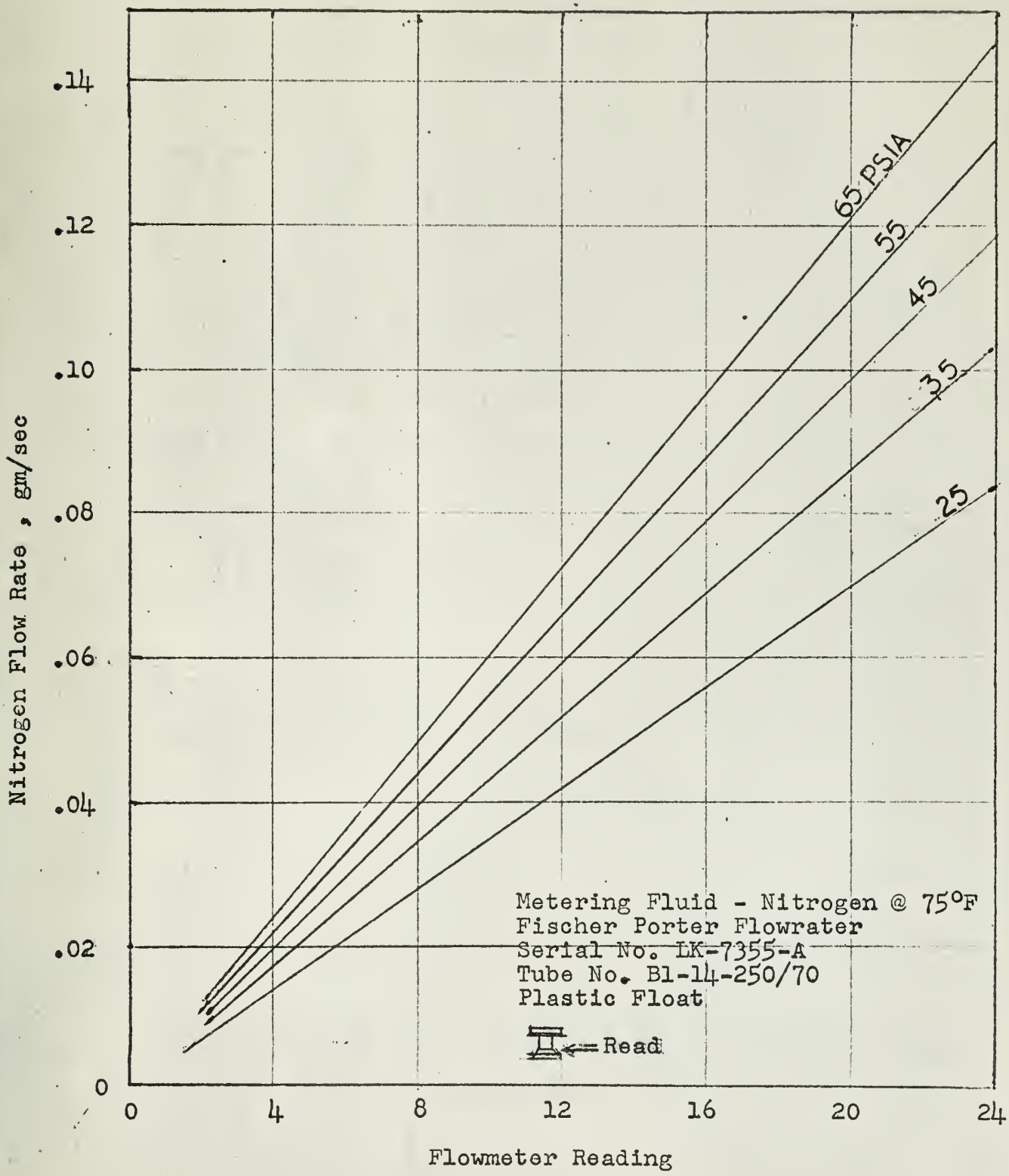
APPENDIX III

SAFETY PRECAUTIONS FOR HANDLING CRYOGENIC FLUIDS

1. Stand clear of boiling or splashing liquid and its issuing gas.
2. When pouring a liquid gas, POUR IT SLOWLY!
3. Insure that a hose or large, open container of water is available to wash off any area of the body that is accidentlly splashed.
4. Never touch any uninsulated pipe or containers with bare hands.
5. Protect eyes with safety goggles or a face shield.
6. Wear loose fitting asbestos or leather gloves.
7. Trousers (cuffless if possible) should be worn outside the shoes.
8. Insure that working area is well ventilated.
9. Never work alone. Evaporating gases may cause the oxygen content of the air to become dangerously low if ventilation is poor.
10. If a person seems to become groggy or loses consciousness while working with liquid nitrogen, get him to a well ventilated area immediately. If breathing has stopped, give artificial respiration. Call a physician at once.

APPENDIX IV

Flowmeter Calibration Curve

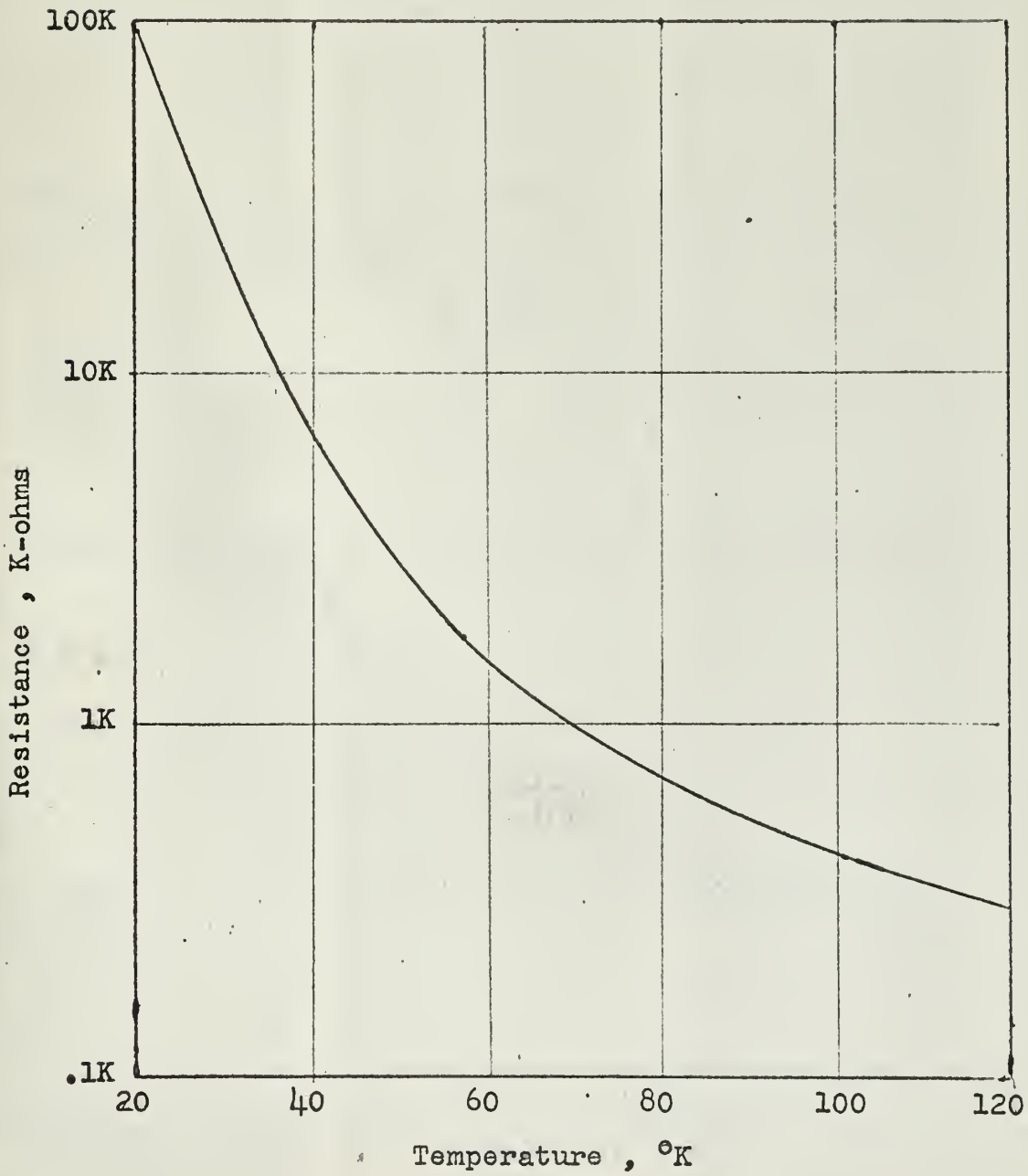


APPENDIX V (a)

Typical Resistance vs. Temperature

Characteristic Curve for

Liquid Nitrogen Range Thermistors

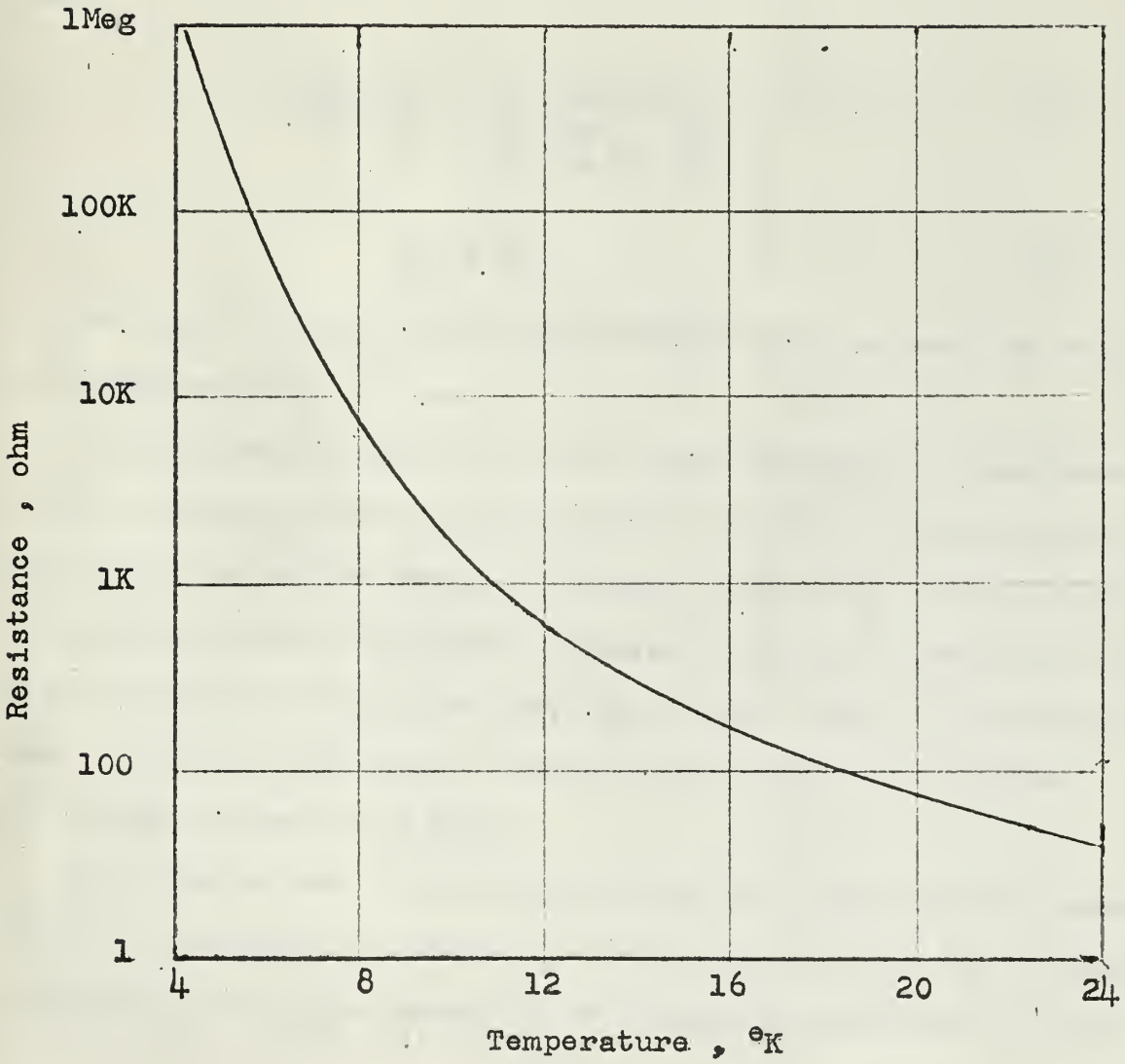


APPENDIX V (b)

Typical Resistance vs. Temperature

Characteristic Curves for

Liquid Helium Range Thermistors



APPENDIX VI

SHIELDING CALCULATIONS

a) Cryopanel.

If the cryopanel, with an emissivity of about .85 and at a temperature of 20°K, is unshielded from the chamber walls, the walls having an emissivity of about .9 and being at a temperature of 300°K, the radiation heat load on the panel may be calculated /13/:

$$q/A_p = \mathcal{F}_{pw}(E_{bw} - E_{bp}) \tag{1}$$

where:

$$\mathcal{F}_{pw}A_p = \frac{1}{\frac{\rho_p}{A_p\epsilon_p} + \frac{1}{F_{pw}A_p} + \frac{\rho_w}{A_w\epsilon_w}} \tag{1a}$$

$$E_b = \sigma T^4 \tag{1b}$$

Solving (1) for q/A_p gives .0389 watts/cm^2 radiation heat load on the unshielded cryopanel.

If a cylindrical shield filled with liquid nitrogen at a temperature of 77°K is placed between the panel and the wall, the emissivity of the shield about .85 and the shield 33" diameter and 36" long, the heat load is similiarly found to be 1.67×10^{-4} watts/cm^2. This is a reduction to .43% of the unshielded radiation heat load on the panel. Thus it is evidenced that a radiation shield greatly reduces the heat load on the cryopanel.

b) Liquid Nitrogen Filled Shield.

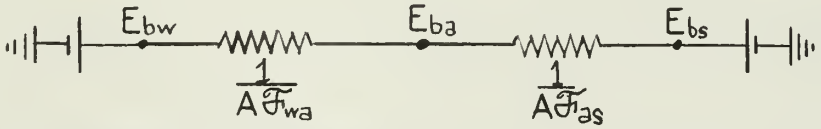
The radiation load on the liquid nitrogen heat shield from the chamber wall may be calculated by assuming the shield and the wall to be flat parallel plates /13/. If this assumption, which neglects end effects, is made,

(1a) reduces to:

$$\mathcal{F}_{ab} = \frac{1}{\frac{1}{\epsilon_a} + \frac{1}{\epsilon_b} - 1} \tag{2}$$

If the emissivity of the chamber walls is taken as .9 and the wall temperature as 300°K, and the emissivity of the outer side of the stainless steel heat shield as .15 and shield temperature as 77°K, the heat load,q/A, may be calculated from (1) to be 67.5×10^{-4} watts/cm^2.

Now let us determine the effect of placing a dry aluminum shield between the wall and the liquid nitrogen shield. Assume the aluminum has an emissivity of .10, on each side. The electrical analog of this is a simple series circuit:



and the heat load may be found:

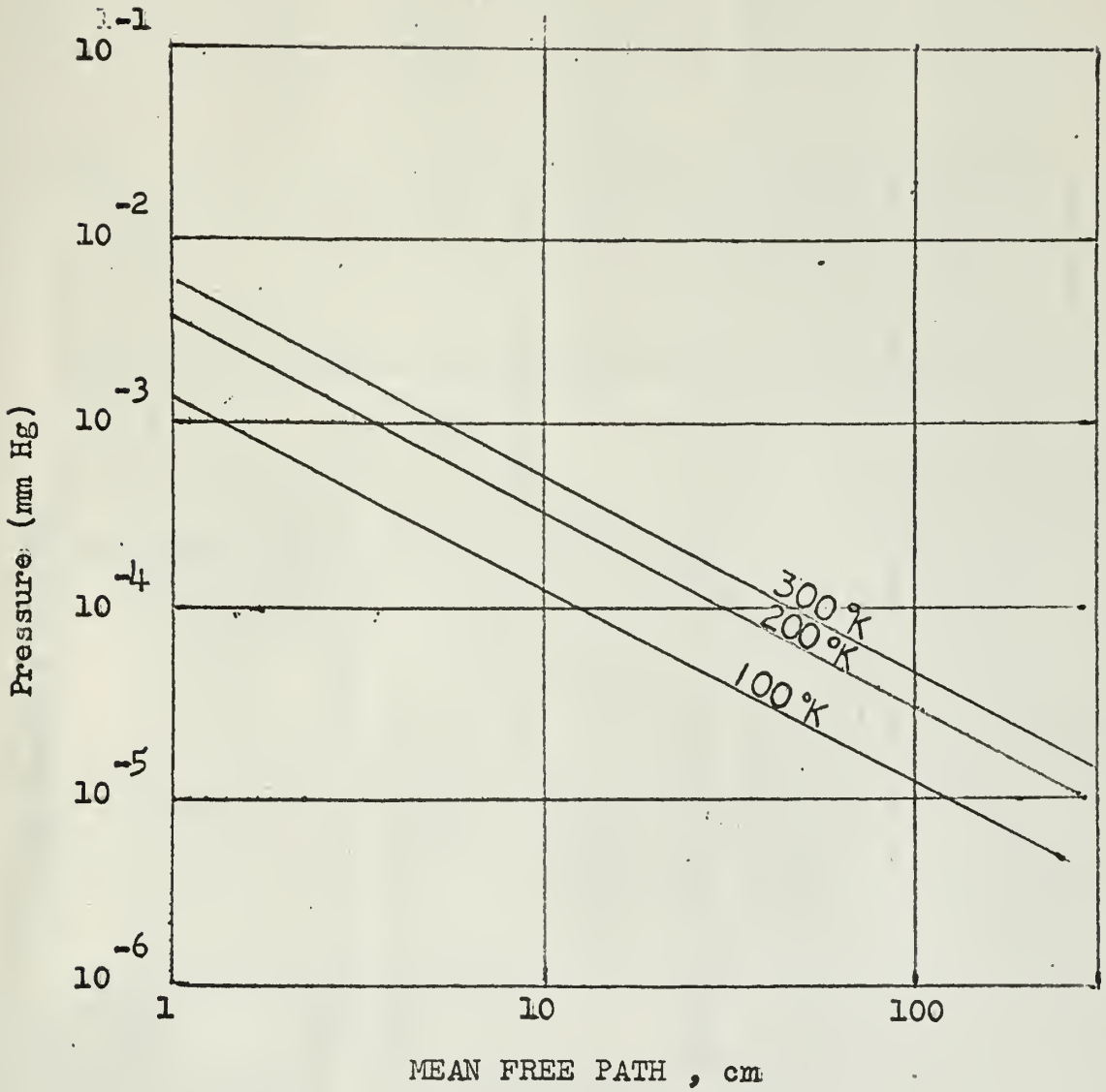
$$\frac{q}{A} = \frac{E_{bw} - E_{bs}}{\frac{1}{\mathcal{F}_{wa}} + \frac{1}{\mathcal{F}_{as}}} \tag{3}$$

Using the above values, q/A is found to be 17.75×10^{-4} watts/cm^2, a reduction to 38% of the heat load on the liquid nitrogen shield without the dry shield.

Thus, a dry shield will substantially reduce the heat load on the liquid nitrogen shield, resulting in less liquid nitrogen requirement and longer operating times per charge.

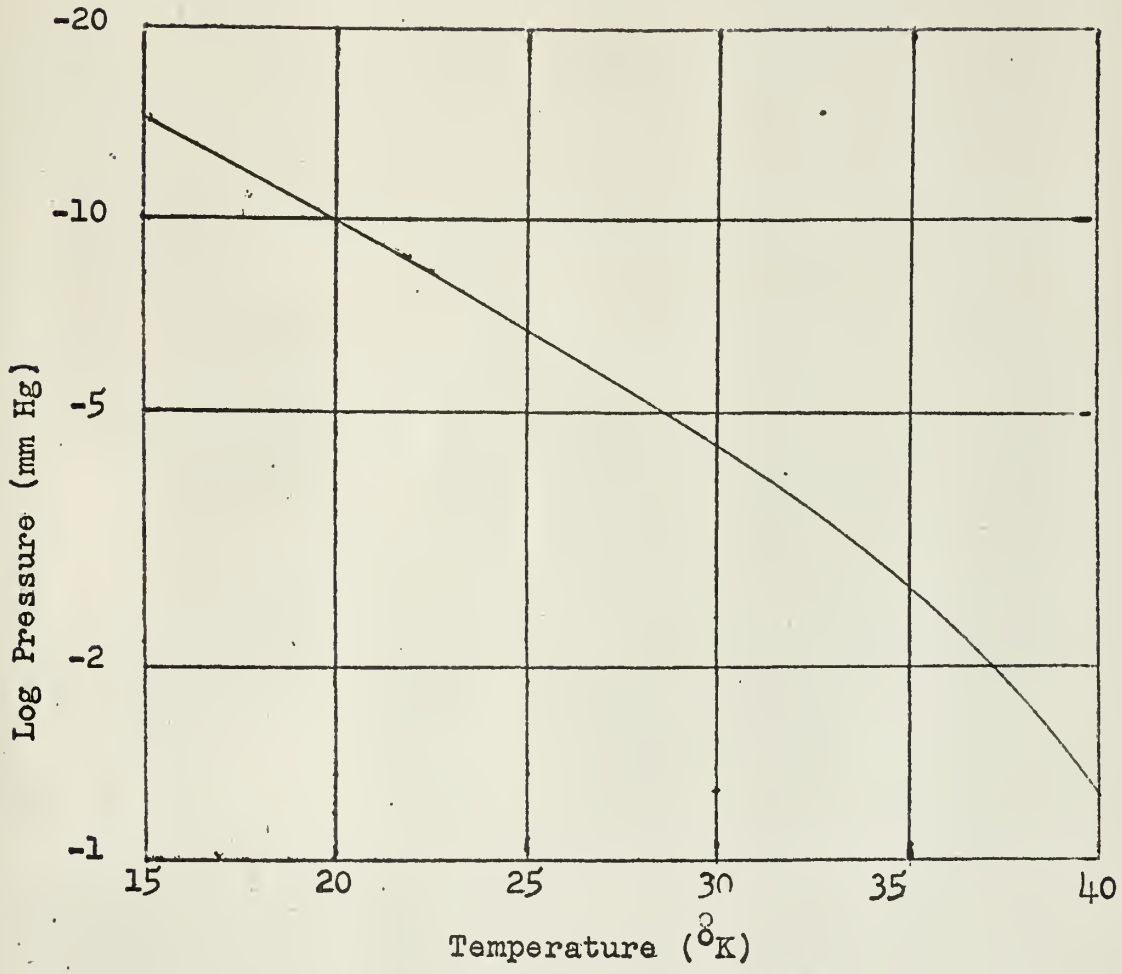
APPENDIX VII

Mean Free Path of Nitrogen vs. Pressure/8/



APPENDIX VIII

Vapor Pressure of Solid Nitrogen vs. Pressure /8/



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